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Indian Standard

SPECIFICATION FOR DIMETHYL TEREPHTHALATE

UDC 547·584

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4

February 1989

Indian Standard

SPECIFICATION FOR DIMETHYL TEREPHTHALATE

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 20 May 1988, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Dimethyl terephthalate (DMT) $[C_6H_4(COOCH_3)_2]$ is normally available in the form of white flakes, pellets or in molten state which is soluble in ether and hot alcohol but sparingly soluble in water. It is derived either by oxidation of *para*-xylene with nitric acid in the presence of methanol or by step-wise catalytic air oxidation of *para*-xylene with intermediate esterification of the *para*-toluic acid.

0.3 DMT is used in the manufacture of polyester resins for the production of film and fibre. It is also used as an intermediate.

0.3.1 The Committee at the time of preparation of this standard envisaged that DMT is also likely to be used for the production of polymer for manufacture of PET bottles in the near future. Cognizance was taken of the fact that an additional requirement of methyl-4-formylbenzoate content would have to be stipulated for DMT used for the manufacture of PET bottles as and when more data is made available.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements, methods of sampling and tests for dimethyl terephthalate.

2. REQUIREMENTS

2.1 The material shall be in the form of white flakes, pellets or in molten state. The molten material shall appear like a colourless liquid having no suspended matter (*see* A-2). It shall be free from any foreign odour, particularly that of amines.

2.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Appendix A. References to the relevant clauses of the Appendix are given in col 4 of the table.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in suitable drums or in any other suitable container as agreed to between the purchaser and the supplier.

3.2 Marking — The containers shall be securely closed after filling and marked with the following:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Net mass of the material in the container; and

d) Lot or batch number.

TABLE 1 REQUIREMENTS FOR DIMETHYL TEREPHTHALATE

(*Clause* 2.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CLAUSE NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Solidification point, °C, *Min*	140·62	A-3
ii)	Colour of melt, Hazen units, *Max*	10	A-4
iii)	Free acidity (as terephthalic acid), percent by mass, *Max*	0·005	A-5
iv)	Volatile matter, percent by mass, *Max*	0·05	A-6
v)	Iron content, ppm, *Max*	1	A-7
vi)	Ash content, ppm, *Max*	10	A-8

3.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clauses* 2.1, 2.2 and *Table* 1)

METHODS OF TEST FOR DIMETHYL TEREPHTHALATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which effect the results of analysis.

A-2. APPEARANCE OF FLAKES, PELLETS AND MELT

A-2.1 Apparatus

A-2.1.1 *Porcelain Dish* — 50-ml capacity.

A-2.1.2 *Pyrex Test Tubes* — *See* A-4.1.1.

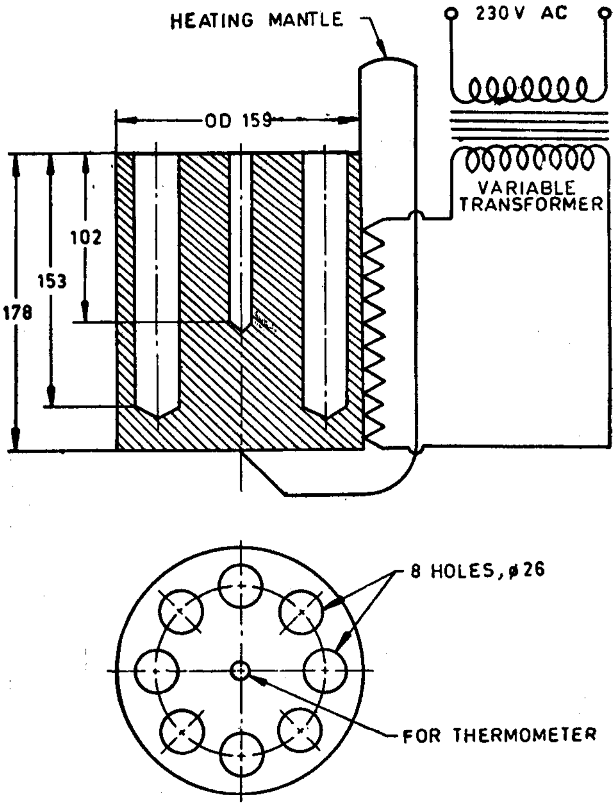
A-2.1.3 *Suitable Light Arrangement*

A-2.1.4 *Aluminium Foil* — 0·03 mm thick for crushing the pellets into powder form.

A-2.1.5 *Hot Aluminium Block* — Capable of maintaining a temperature of 175 ± 1 °C through thermostat control (*see* Fig. 1).

NOTE 1 — These blocks are cylindrical in shape with eight holes of suitable diameter and depth drilled from the top surface. One pocket for thermometer and one for thermostat element is provided. The block is enclosed with heating mantle except on top and bottom surface and well insulated except on top surface. Depending on the required number of holes for test tube, the size of the block may be modified suitably. A sketch for typical hot aluminium block is given in Fig. 1.

NOTE 2 — Oil bath of suitable dimensions maintained at 175 ± 1 °C may also be used. Oils generally become discoloured on prolonged heating. Any oil adhering to the test tubes has to be removed prior to colour test (*see* A-4).



All dimensions in millimetres.

FIG. 1 A SKETCH FOR HOT ALUMINIUM BLOCK FOR COLOUR TEST OF DMT

A-2.2 Procedure — The appearance of dimethyl terephthalate is judged visually. The flakes and pellets are viewed by reflected light in a porcelain dish. The flakes are melted in a pyrex tube directly while pellets are first crushed to powder form in aluminium foil prior to melting in pyrex tube.

---

*Specification for water for general laboratory use (*second revision*).

The melt shall be colourless and free from suspended solids.

A-3. DETERMINATION OF SOLIDIFICATION POINT

A-3.1 Determine the solidification point of DMT according to the method prescribed in IS : 5813-1970*. The thermometer used for the purpose shall be of the range of 139 to 143°C with graduation of 0·02°C.

A-4. TEST FOR COLOUR

A-4.0 Outline of the Method — The colour of the molten material is compared with that of the colour standard and expressed in terms of Hazen colour units. One Hazen colour unit is defined as the colour of an aqueous solution containing 1 ppm of platinum in the form of chloroplatinic acid and 2 ppm of cobaltous chloride ($CoCl_2 . 6 H_2O$).

A-4.1 Apparatus

A-4.1.1 *Pyrex Test Tubes* — with the following dimensions:

a)	Outer dia	: 25 mm
b)	Overall height	: 200 mm
c)	Wall thickness	: 1·2 to 1·8 mm
d)	Thickness of bottom	: 1·5 to 3·0 mm

A-4.1.2 *One-Mark Volumetric Flasks* — 250-ml and 500-ml capacities (*see* IS : 915-1975†).

A-4.1.3 *Hot Aluminium Block* — *See* **A-2.1.5.**

A-4.2 Reagents

A-4.2.1 *Cobaltous Chloride Hexahydrate* — Solid.

A-4.2.2 *Hydrochloric Acid* — Relative density 1·16 (*see* IS : 265-1977‡).

A-4.2.3 *Chloroplatinic Acid* — Dissolve 250 mg of platinum in small quantity of aqua regia contained in a glass or porcelain basin by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Repeat this operation twice more.

A-4.3 Preparation of Colour Standard — Dissolve 0·50 g of cobaltous chloride hexahydrate and entire quantity of the chloroplatinic acid in 50 ml of hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into 500-ml one-mark volumetric flask. Dilute with water to the mark.

A-4.3.1 Pipette 5 ml of the solution (**A-4.3**) into a 250-ml one-mark volumetric flask and dilute with water to the mark. This diluted solution has colour of 10 Hazen units, and should always be prepared freshly.

A-4.4 Procedure — If the sample is in the form of flakes, transfer about 40 g, portion by portion into the test tubes kept in hot aluminium block maintained at 175 ± 1°C (**A-4.1.3**). In case the sample is in the form of pellets or lumps, wrap sufficient pellets (40 g) in an aluminium foil (**A-2.1.4**) and crush into powder.

When the material is well melted, withdraw the test tubes from the aluminium block, hold them slanting at 45° against evenly lighted white background and compare quickly the colour against the colour standard (**A-4.3**) kept in a separate test-tube filled to the same height.

NOTE — Colour comparison by viewing vertically downwards is difficult owing to condensation of DMT crystals at upper cooler portion of the test tubes.

A-4.4.1 The material shall be taken to have passed the test if the intensity of colour of the material is not more than that of the colour standard.

A-5. DETERMINATION OF FREE ACIDITY

A-5.0 Outline of the Method — Free acidity is determined as percent of terephthalic acid. A suitable amount of sample is dissolved in chloroform and free acid is titrated with alcoholic potassium hydroxide under inert atmosphere to phenolphthalein end point.

A-5.1 Apparatus

A.5.1.1 *Microburette* — 2 × 0·01 ml.

A-5.1.2 *Conical Flask* — 250 ml, equipped with rubber stopper with holes for burette and nitrogen sparge tube (*see* Fig. 2).

A-5.1.3 *Magnetic Stirrer and Stirring Bar*

A-5.1.4 *Sparge Tube* — For nitrogen (*see* Fig. 2).

A-5.2 Reagents

A-5.2.1 *Chloroform* — *See* IS : 5296-1979*.

A-5.2.2 *Standard Alcoholic Potassium Hydroxide Solution* — 0·01 N.

A-5.2.3 *Phenolphthalein* — 1 percent solution (*see* IS : 2263-1979†).

A-5.2.4 *Dry Nitrogen Gas*

---

*Methods for determination of crystallizing point.

†Specification for one-mark volumetric flasks.

‡Specification for hydrochloric acid (*second revision*).

*Specification for chloroform, pure and technical (*first revision*).

†Methods of preparation of indicator solutions (*first revision*).

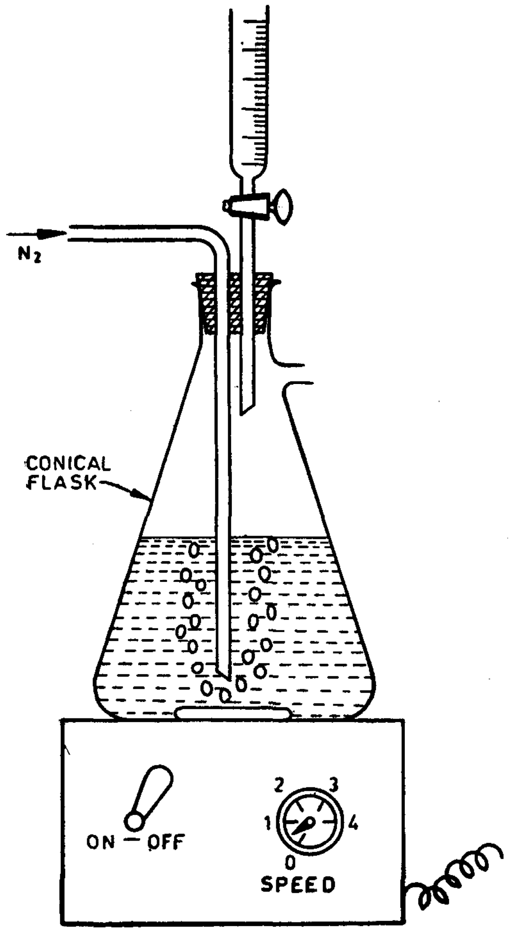


FIG. 2 ASSEMBLY OF CONICAL FLASK WITH BURETTE AND NITROGEN SPARGE TUBE

A-5.3 Preparation of Sample

A-5.3.1 *Flakes* — Use as such.

A-5.3.2 *Pellets* — Wrap requisite number of pellets in an aluminium foil and beat into fine powder, mix well.

A-5.3.3 *Molten DMT* — Pour sufficient sample on to aluminium foil. Allow to solidify completely. Beat into fine powder as in **A-5.3.2.**

A-5.4 Procedure — Assemble the apparatus as shown in Fig. 2. Introduce 100-150 ml chloroform in conical flask. Start stirring and nitrogen sparge. After about five minutes, add 8-10 drops of phenolphthalein and titrate with 0·01 N KOH to first appearance of a permanent pink colour. Set the burette to zero reading. Add about 20 g of powdered homogenized sample, weighed accurately up to 0·1 g to conical flask. Continue stirring and nitrogen sparge. When sample is completely dissolved, titrate with 0·01 N KOH until the first appearance of a permanent pink colour. Note the burette reading (V).

A-5.5 Calculation

Free acidity, as terephthalic acid,

$$\text{percent by mass} = \frac{V \times N \times 8{\cdot}306}{M}$$

where

V = volume in ml of standard alcoholic potassium hydroxide solution,

N = normality of standard alcoholic potassium hydroxide solution, and

M = mass in g of the sample taken for the test.

A-6. DETERMINATION OF VOLATILE MATTER

A-6.0 General — Volatile matter is the difference in mass of the untreated sample and the sample treated under specified conditions.

A-6.1 Apparatus

A-6.1.1 *Glass Tray* — of capacity 300-ml.

A-6.1.2 *Desiccator*

A-6.1.3 *Vacuum Pump*

A-6.2 Procedure — Weigh 100 g of the material into a glass tray. Keep the tray in a desiccator containing calcium chloride and expose the sample for 12 hours in room temperature to a vacuum of 1·3 kN/m^2 to 2·6 kN/m^2. Reweigh to determine the loss in mass.

A-6.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{(M_1 - M_2) \times 100}{M_1}$$

where

M_1 = mass in g of the material taken for the test, and

M_2 = mass in g of the material after drying.

A-7. DETERMINATION OF IRON CONTENT

A-7.0 Outline of the Method — Pure dimethyl terephthalate is incinerated and the iron contained in the ash is converted into iron thioglycollate. The concentration is determined photometrically.

A-7.1 Apparatus

A-7.1.1 *Spectrophotometer*

A-7.2 Reagents

A-7.2.1 *Thioglycollic Acid Solution* — 10 percent. If after its preparation, the solution becomes turbid, it should be filtered.

A-7.2.2 *Sodium Citrate Solution* — Dissolve 150 g of AR grade trisodium citrate in water and make up to 500 ml. Transfer this solution into a 1 000 ml separating funnel, add 0·5 ml of concentrated ammonium hydroxide solution and 50 ml of 0·02 percent dithiozone in chloroform solution, and shake well. After phase separation, extract the aqueous phase several times, using 50 ml of diluted dithiozone solution (10 ml of 0·02 percent dithiozone solution and 40 ml of chloroform for each extraction). Then add 5 ml of 20 percent citric acid solution and shake with 50 ml of chloroform each time until the final extract is colourless. Store the solution in a polyethylene bottle.

A-7.2.3 *Standard Iron Solution* — Dissolve 0·702 3 g of pure ammonium ferrous sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in water contained in a 1 000-ml measuring flask. Add 5 ml of sulphuric acid and dilute the contents to 1 000 ml.

Further dilute 25 ml of this solution to 250 ml. 1 ml of prepared solution corresponds to 10 μg of Fe.

A-7.3 Preparation of Calibration Chart — Fill a set of 50-ml measuring flasks with 2·0, 4·0, 8·0, 12·0, 16·0 and 20·0 ml of standard iron solution and add 5 ml of dilute sulphuric acid into each flask.

Further add 2 ml of sodium citrate solution and dilute the contents with water to 25 to 27 ml. Add 5 ml of concentrated hydrochloric acid and while cooling the solution, add concentrated ammonium hydroxide solution drop by drop until a slightly alkaline reaction is obtained. Now add another 1·5 ml of hydrochloric acid in excess.

Cool the contents well and mix 1·0 ml of 10 percent thioglycollic acid solution, then add 3·0 ml of concentrated ammonium hydroxide solution and make up the solution to 50 ml.

The reference solution is prepared in the same manner but without the iron solution.

After the solutions have been allowed to stand for 5 minutes, measure the absorption of each solution in a 4 cm-cell at a wavelength of 535 nm, using the iron-free solution for reference. On the basis of the values obtained, prepare a chart by plotting the iron content in μg as a function of the measured absorption.

A-7.4 Procedure — Incinerate 30 g of the material in a quartz crucible. After the flame is extinguished, transfer the crucible to a muffle furnace maintained at 550°C and continue incineration for 30 minutes. Allow it to cool and dissolve the contents in 10 ml of hydrochloric acid solution (1 : 1) by adding through the sides of the dish. Bring it to boil on a hot plate. Transfer it to 50 ml measuring flask. Wash with distilled water three times and transfer to the measuring flask. Add 2 ml of sodium citrate solution followed by 1 ml of 10 percent thioglycollic acid solution. Add excess of ammonia solution to ensure that the solution remained alkaline. Make up to the mark with distilled water and mix thoroughly. At the end of 5 minutes, measure the absorption at 535 nm using the spectrophotometer.

A-7.5 Calculations

$$\text{Iron content, parts per million} = \frac{A}{M}$$

where

A = iron content in μg as read from the chart, and

M = mass of the sample taken for the test.

NOTE 1 — If the iron concentration is low, extreme care is taken to prevent foreign matter from getting into the sample during the incineration.

NOTE 2 — If the concentration is so high that on comparison with the 4 cm-cell the linear range is exceeded, measurements should be repeated using a diluted solution.

A-8. DETERMINATION OF ASH

A-8.0 Outline of the Method — Ash is determined as the residue left after ignition of a known mass of the sample and finally heated in a burner.

A-8.1 Apparatus

A-8.1.1 *Muffle Furnace*

A-8.1.2 *Blast Burner*

A-8.1.3 *Platiuum Dish*

A-8.1.4 *Micro Balance*

A-8.1.5 *Crucible Tongs* — of stainless steel with platinum tips.

A-8.1.6 *Glazed Porcelain Plates*

A-8.2 Procedure — Clean the platinum dish free from grease and salts by soaking in hot chromic acid washing with HCl (1 : 1) followed by distilled water. Place the dish in the muffle furnace with the help of platinum tipped crucible tongs and heat at 550°C for 30 minutes. Remove the dish from the furnace and place it on a clean porcelain plate which is free of dust particles. Allow the dish to cool for one minute and transfer it to a desiccator which is placed near the micro balance. Weigh the empty dish and again with sample of the material to nearest 0·1 g. Place the dish with the material on a triangle supported by a tripod and ignite the sample by means of a Maker burner (*see* Note 1). After ignition, place the dish in the muffle furnace at 550°C and remove the same from the furnace when all carbon is removed (*see* Note 2). Place the dish with ash in the desiccator for 30 minutes. Weigh the dish with its contents.

NOTE 1 — A minimum of 500 g of sample is to be incinerated.

NOTE 2 — Once the sample has been reduced to carbonaceous ash, extreme care must be exercised in handling the dish. If the dish is handled in the usual manner with the tongs, carbon may be transferred to the tips of the tongs and may add carbon to the ash when the dish is removed from the furnace. In order to prevent this source of serious error, tongs may be turned with the tips pointing upwards and the dish may be picked up by placing the arcs of the tongs around the body of the dish. The dish should not make contact with the refractory lining of the furnace as particles of platinum may be scratched off thus changing its mass.

A-8.3 Calculations

$$\text{Ash, ppm} = \frac{M_3 - M_1}{M_2 - M_1} \times 1\,000\,000$$

where

M_1 = mass in g of empty dish,

M_2 = mass in g of empty dish with sample, and

M_3 = mass in g of dish with ash.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF DIMETHYL TEREPHTHALATE

B-1. GENERAL REQUIREMENTS FOR SAMPLING

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action. In case of molten DMT, sample shall be drawn in a clean, dry stainless steel thermos flask. The contents are then poured carefully in clean, dry aluminium petri dishes and allowed to cool. The solidified lumps are then placed in clean, dry air-tight bottles or suitable containers.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and the details given under **3.2**.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot.

B-2.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, samples shall be tested for each lot separately.

B-2.3 The number of containers to be selected from the lot for this purpose shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE (1)	NUMBER OF CONTAINERS TO BE SELECTED (2)
Up to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

B-2.3.1 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedures given in IS : 4905-1968* may be followed.

B-3. TEST SAMPLES AND REFEREE SAMPLES

B-3.1 From each of the containers selected according to **B-2.3**, a representative portion of the material shall be drawn.

B-3.2 From each of these individual portions (*see* **B-3.1**), a small but approximately equal quantity of material shall be taken and thoroughly mixed to constitute a composite sample of not less than 1 500 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and third for the referee.

---

*Methods for random sampling.

B-3.3 The referee sample consists of the composite sample marked for this purpose, and shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the two and shall be used in case of dispute.

B-4. NUMBER OF TESTS

B-4.1 Tests for determination of all the requirements given in this specification shall be carried out on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to the requirements of this specification if all the test results on the composite sample meet the corresponding specification requirements.

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AMENDMENT NO. 1 AUGUST 2002
TO
IS 12438 : 1988 SPECIFICATION FOR DIMETHYL TEREPHTHALATE

(*Page* 1, *clause* **1.1**) — Insert the following new clause after **1.1** and renumber the subsequent clauses:

'2 REFERENCES

The following standards contain provision which through reference in the text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
2362 : 1993	Determination of water by the Karl Fischer method — Test method (*first revision*)
13320 : 1992	Determination of iron by atomic absorption spectrophotometry — Test method'

[*Page* 1, *Table* 1, *Sl No.* (iii)] — Substitute the following for the existing:

'(1)	(2)	(3)	(4)
iii)	Acid number (mg, KOH/g)	0.03	A-5'

[*Page* 1, *Table* 1, *Sl No.* (iv)] — Substitute the following for the existing:

'(1)	(2)	(3)	(4)
iv)	Moisture content, percent by mass, *Max*	0.02	IS 2362 : 1993'

(*Page* 4, *clause* **A-5.5**) — Substitute the following for the existing formula:

$$\text{Acid number (mg, KOH/g)} = \frac{V \times N \times 56.1}{M}$$

(*Page* 4, *clause* **A-6**) — Delete.

Gr 1

(*Page* 4, *clause* A-7) — Substitute the following for the existing clause:

A-7 DETERMINATION OF IRON CONTENT

A-7.0 General

Two methods have been specified for determination of iron content, namely:

a) Method 1 — Photometric method; and

b) Method 2 — Atomic absorption spectrophotometric method.

Both the methods can be used for determination of iron on routine basis. However in the event of any dispute, Method 2 should be treated as a referee method.

A-7.1 Method 1 : Photometric Method

A-7.1.1 *Outline of the Method*

Pure dimethyl terephthalate is incinerated and the iron contained in the ash is converted into iron thioglycollate. The concentration is determined photometrically.

A-7.1.2 *Apparatus*

A-7.1.2.1 *Spectrophotometer*

A-7.1.3 *Reagents*

A-7.1.3.1 *Thioglycollic acid solution* — 10 percent. If after its preparation, the solution becomes turbid, it should be filtered.

A-7.1.3.2 *Sodium citrate solution* — Dissolve 150 g of AR grade trisodium citrate in water and make up to 500 ml. Transfer this solution into a 1 000-ml separating funnel, add 0.5 ml of concentrated ammonium hydroxide solution and add 50 ml of 0.02 percent dithiozone in chloroform solution, and shake well. After phase separation, extract the aqueous phase several times, using 50 ml of diluted dithiozone solution (10 ml of 0.02 percent dithiozone solution and 40 ml of chloroform for each extraction). Then add 5 ml of 20 percent citric acid solution and shake with 50 ml of chloroform each time until the final extract is colourless. Store the solution in a polyethylene bottle.

A-7.1.3.3 *Standard iron solution* — Dissolve 0.702 3 g of pure ammonium ferrous sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in water contained in a 1 000-ml measuring flask. Add 5 ml of sulphuric acid and dilute the contents to 1 000-ml.

Further dilute 25 ml of this solution to 250 ml. 1 ml of prepared solution corresponds to 10 µg of iron.

A-7.1.3.4 *Preparation of calibration chart* — Fill a set of 50-ml measuring flasks with 2.0 ml, 4.0 ml, 8.0 ml, 12.0 ml, 16.0 ml and 20.0 ml of standard iron solution and add 5 ml of dilute sulphuric acid into each flask.

Further add 2 ml of sodium citrate solution and dilute the contents with water to 25 to 27 ml. Add 5 ml of concentrated hydrochloric acid and while cooling the solution, add concentrated ammonium hydroxide solution drop-by-drop until a slightly alkaline reaction is obtained. Now add another 1.5 ml of hydrochloric acid in excess.

Cool the contents well and mix 1.0 ml of 10 percent thioglycollic acid solution, then add 3.0 ml of concentrated ammonium hydroxide solution and make up the solution to 50 ml.

The reference solution is prepared in the same manner but without the iron solution.

After the solutions have been allowed to stand for 5 minutes, measure the absorption of each solution in a 4-cm cell at a wavelength of 535 nm, using the iron-free solution for reference. On the basis of the values obtained, prepare a chart by plotting the iron content in µg as a function of the measured absorption.

A-7.1.3 .5 *Procedure* —Incinerate 30 g of the material in a quartz crucible. After the flame is extinguished, transfer the crucible to a muffle furnace maintained at 550°C and continue incineration for 30 minutes. Allow it to cool and dissolve the contents in 10 ml of hydrochloric acid solution (1:1) by adding through the sides of the dish. Bring it to boil on a hot plate. Transfer it to 50-ml measuring flask. Wash with distilled water three times and transfer to the measuring flask. Add 2 ml of sodium citrate solution followed by 1 ml of 10-percent thioglycollic acid solution. Add excess of ammonia solution to ensure that the solution remained alkaline. Make up to the mark with distilled water and mix thoroughly. At the end of 5 minutes, measure the absorption at 535 nm using the spectrophotometer.

A-7.1.4 *Calculations*

$$\text{Iron content, parts per million} = \frac{A}{M}$$

where

A = iron content in µg as read from the chart, and

M = mass of the sample taken for the test.

NOTES

1 If the iron concentration is low, extreme care is taken to prevent foreign matter from getting into the sample during the incineration.

2 If the concentration is so high that on comparison with the 4-cm cell the linear range is exceeded, measurements should be repeated using a diluted solution.

A-7.2 Method 2 : Atomic Absorption Spectrophotometric Method

A-7.2.1 *Outline of the Method*

About 100 g of DMT in powdered form is sublimed in platinum dish using hot plate. A platinum dish with unsublimed content is incinerated in muffle furnace. Ash obtained is dissolved in dilute hydrochloric acid and made up to suitable volume. The solution thus obtained is used for determination of iron using atomic absorption spectrophotometer (*see* also IS 13320 : 1992).

A-7.2.2 *Apparatus*

A-7.2.2.1 *Platinum dish* — 50-ml capacity.

A-7.2.2.2 *Muffle furnace*

A-7.2.2.3 *Hot plate*

A-7.2.2.4 *Micro balance*

A-7.2.2.5 *Atomic absorption spectrophotometer*

A-7.2.3 *Reagents*

A-7.2.3.1 *Hydrochloric acid solution* — (1:1 V).

A-7.2.3.2 *Iron Standard 3 ppm* — Dissolve 0.021 1 g of pure ammonium ferrous sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in distilled water. Add 5 ml of 1:1 HCl and dilute to 1 000 ml with distilled water.

A-7.2.4 *Procedure* — Weigh about 100 g of powdered DMT (S g) and put small quantity of it in clean platinum dish. Place platinum dish on hot plate and allow DMT to sublime in a fume cupboard. Repeat with remaining quantity of DMT till the entire weighed sample is consumed. Transfer platinum dish to muffle furnace at 550°C for 15 minutes. Remove the dish from furnace and allow it to cool in a desiccator. Dissolve the contents in 10 ml (1:1) hydrochloric acid and warm it using hot plate. Transfer it to 25-ml volumetric flask. Make up the volume by rinsing platinum dish with distilled water. Prepare a blank in 25-ml volumetric flask by taking 10 ml hydrochloric acid (1:1) and make up the volume with distilled water. Analyze iron using atomic absorption spectrophotometer.

A-7.2.5 *Calculation*

$$\text{Iron, ppm} = \frac{\text{Absorbance of sample}}{\text{Absorbance of standard}} \times 3 \times \frac{25}{S}$$

where

S = mass of DMT sample in g.

(*Page* 5, *clause* **A-8.2**, *line* 13) — Substitute 'to the nearest 0.1 mg' *for* 'to nearest 0.1 g'.

(PCD 9)

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